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MOLECULAR BEAM EPITAXIAL GROWTH AND ELECTRICAL AND  
OPTICAL INVESTIGATIONS (U) UNIVERSITY OF SOUTHERN  
CALIFORNIA LOS ANGELES DEPT OF AEROSPA. A MADHUKAR

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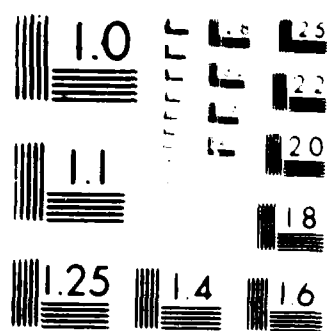
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**FINAL SCIENTIFIC REPORT**

**AFOSR GRANT NO. 83-0354**

**TITLE: MOLECULAR BEAM EPITAXIAL GROWTH AND  
ELECTRICAL AND OPTICAL INVESTIGATIONS OF  
III-V COMPOUND SEMICONDUCTORS**

**PERIOD: SEPT.1, 1983 - FEB. 28, 1985**

**PRINCIPAL INVESTIGATOR: ANUPAM MADHUKAR  
UNIVERSITY OF SOUTHERN CALIFORNIA  
LOS ANGELES, CA 90089-0241**

**ABSTRACT**

This grant provided funds under the University Research Instrumentation Program (URIP) for the acquisition of capital equipment for research in the area of molecular beam epitaxial growth of III-V semiconductor based single and multiple interface quantum well structures and their characterization via optical and electrical methods. This report provides a summary of the activities relating to acquisition of the equipment and a list of all the equipment acquired.

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The AFOSR grant number 83-0354 provided funds under the University Research Instrumentation Program to acquire equipment for seven different specific scientific tasks comprising our overall scientific work on the molecular beam epitaxial (MBE) growth and characterization of GaAs/Al<sub>x</sub>Ga<sub>1-x</sub>As single and multiple interface structures. As such, this report is broken into seven sections, each providing a summary of the activity in a given area.

#### A. SUBSTRATE PREPARATION FACILITIES:

To establish the basic facilities needed for preparation of GaAs substrates used in MBE growth, equipment was acquired for

- (A.1) an ultrahigh purity (UHP) de-ionized (DI) water system.
- (A.2) handling of source charge materials, chemical reagents, etc.
- (A.3) maintaining laminar flow clean environment during mounting of the substrates, etc.

A schematic diagram of the UHP-DI water system is shown in fig. 1 and was designed by us. Considerable time was spent in both designing the system to achieve high quality, semiconductor processing grade water (resistivity  $\geq 18$  M, total organic content  $\leq 500$  parts per billion, molecular weight cut-off of 10,000) and identifying individual components and vendors capable of delivering and installing such a system. A certain element of unexpected surprise in achieving this objective had to be faced since close examination of standard systems suggested by the vendors (supposedly supplying such equipment and systems to semiconductor industry) reflected many gaps in the overall comprehension of the interplay between various components of such a system. Lack of some basic considerations of significance in choosing the appropriate piping material, types of grease, joints, etc. by the installers also led to the unanticipated need for constant supervision by us during the installation phase. Nevertheless, with a delay of some six months due to the above noted reasons, the system was installed and made operational by late 1983 and has, with proper maintenance, performed quite well ever since.

A fume hood and a laminar flow bench were installed along with a sink, as also shown schematically in fig. 1. Outlets for DI water are provided in the fume hood and the laminar flow hood. The latter is also equipped with rinse tanks with continuous flow of the DI water. The points of delivery with respect to the filtering and water return system are placed in such a manner that the highest purity water is obtained in the laminar flow bench.

## B. MODIFICATION OF THE $\phi$ -400 MBE SYSTEM:

The source oven design and the pumping system on the MBE growth chamber in the Perkin-Elmer ( $\phi$ ) 400 system were changed. The former was needed to achieve greater control and stability in the flux through improved thermocouple based temperature detection and feed-back control system and the latter to achieve effective pumping of the growth chamber, particularly during system bakeout. To achieve the first objective, the shape of the PBN crucible was changed from cylindrical without any lip to tapered with lip, along with the thermocouple being spot welded to a tantalum strip surrounding the crucible at the lower end. Originally, the thermocouple merely relied upon gravity to achieve mechanical contact with the crucible, thus leading to poor contact as well as a mechanically unstable system. Many different variations of the new conceptual approach outlined above were implemented and tested before settling on the specific system in operation since fall of 1984.

A pumping well with a Ti ball sublimator, internal cryoshrouding and a chevron on top was designed to be interfaced with the growth chamber. The pumping well allows for horizontal mounting of an ion pump from one side and a cryopump from the other. In the original  $\phi$ -400 system only a cryopump was mounted on the growth chamber and that too vertically looking up with no chevron or  $\text{LN}_2$  baffling. In this configuration it loses its efficiency rather rapidly due to contamination by arsenic flakes and powder, and cannot stand the thermal radiation load during bakeout. The new pumping configuration avoids both these problems and in addition provides a TSP and an ion pump for direct pumping of the growth chamber. A schematic drawing of the system fabricated is shown in fig. 2.

Finally, a computer system (MicroVAX II) along with the appropriate interfacing accessories (A/D and D/A convertors, printer, etc.) were acquired in spring of 1985 for automation of the MBE system. Considerable work on the development of the software and testing of many of these operations has been done during this time. However, the success of our MBE growth efforts and the critical timing of these efforts vis a vis the fast moving literature of basic RHEED intensity dynamics studies (see next section), as well as growth employing the new notion of interrupted growth introduced by us were such that we decided not to shut down the operations for actually implementing the computer interfacing. We expect to take this step in the near future.

### C. RHEED INTENSITY MEASUREMENT SYSTEM:

Reflection-high-energy-electron-diffraction (RHEED) has been an integral part of most MBE growth chambers and has been customarily employed as a diagnostic tool for identification of the surface reconstruction conditions of the starting surface and during growth. Around 1982 it was realized that the intensity of the various features of the RHEED pattern shows a damped oscillatory temporal behavior during growth with the oscillation period coinciding with the monolayer growth time under the usual growth conditions employed for GaAs homo-epitaxy. This discovery coincided with our attempts at computer simulations of the MBE growth process which not only also suggested the existence of this phenomenon, but more importantly held the promise of correlating the observed behavior with the growth condition controlled surface kinetic processes responsible for determining the dynamics of the growth front morphology (step density distribution). We therefore felt at the time that RHEED could be established as a meaningful real-time, in-situ, research tool for gaining insight into the nature of the MBE growth kinetics, as well as a pragmatic tool for identifying optimized growth conditions for achieving high quality interfaces. Motivated by such considerations, we undertook the task of designing a simple, economical and practically useful system for measurement of the RHEED intensity dynamics. A schematic diagram of the system we built with equipment purchased through funds provided under this grant is shown in fig. 3.

While the intensity measurement system is the simplest system based upon a Si photodetector, a unique and useful feature of this system is the use of a low light sensitive TV camera and a video recorder system to record the entire RHEED pattern and the time dependent behavior of every feature for future detailed and quantitative analysis without the need for repeating the experiment. Through the use of a TV monitor, a particular RHEED feature, generally the specular spot, of interest during the actual growth is monitored through the detection system for real-time monitoring of growth. Another TV monitor allows visual monitoring of the full RHEED pattern to ensure that with the variation of the growth conditions (substrate temperature, group V pressure, and group III flux) the surface reconstruction, etc., remain as desired. This system has been in operation since fall 1983 and has produced many exciting results reported in a number of publications from our group. Indeed, information regarding this system has been sought by many professional colleagues and has been provided to them over the past three years. The Air Force Avionics Laboratory at Wright Patterson (Dr. K.R. Evans), Dept. of Electrical Engineering at U.C. Santa Barbara (Prof.

H. Kroemer), AT&T Bell Labs. (Dr. A.Y. Cho) and Naval Weapons Center, China Lake, California (Dr. V. Rehn) are some of the places who have requested this information.

#### D. OPTICAL MEASUREMENTS:

A cryostat capable of achieving temperatures down to 3°K and with appropriate optical windows for measurement of photoluminescence (PL), PL excitation (PLE) spectra, and optical absorption behavior of quantum well structures was acquired, along with some of the needed accessories (such as pumping system, optical table, light source and detection system, etc.). This represented a re-ordering of our priorities since originally it was planned to acquire the cryostat with a superconducting magnet ( $\leq 7$  Tesla) for magneto-transport. The superconducting magnet was sacrificed in the interest of acquiring part of the optical instrumentation needed for the PL, PLE and absorption measurements noted above. The reordering of the priorities of the types of measurements itself was made on consideration of two evolving factors - (i) the lack of adequate in-house capabilities for deposition of appropriate and reliable metal contacts essential for high field magneto-transport measurements but not needed for optical measurements, and (ii) the greater suitability of PL and PLE measurements and ease of interpretation of the results in relation to their ability to provide information on the nature of GaAs/Al<sub>x</sub>Ga<sub>1-x</sub>As interfaces realized on the basis of our RHEED intensity determined growth conditions. This system, shown schematically in fig. 4, has been operational for the past two years and has led to publication of many systematic studies of the inter-relationship between RHEED intensity dynamics, the underlying kinetics of MBE growth, and the atomistic structural and chemical nature of high quality interfaces as probed in optical experiments.

#### E. NEAR-IR SPECTROSCOPIC ELLIPSOMETRY:

Funds were requested and provided under this grant to extend the existing variable wave length (spectroscopic) ellipsometry set-up from its UV-visible range into the near IR range (out to  $\sim 2.5 \mu\text{m}$ ). To this end, a thermoelectrically cooled PbS detector, a single pass monochromator, and other appropriate accessories were acquired by mid 1984 and the task of extending the fully automated ellipsometry system begun. All the necessary computer interfacing and software development was completed and tested by end of 1985. Some unexpected delays occurred since the LSI 11/23 computer system controlling the ellipsometry instrumentation developed problems which the DEC field engineers could not fix for over five months. Finally, our own efforts solved the problem and allowed not



only continuation of research in the UV-visible range, but also initiation of measurements into the near IR regime. Many difficulties with acquiring good enough signal to noise, however, have continually been faced during this past year. Considerable improvement in signal to noise ratio has been achieved through a variety of "light-saving" measures implemented, but it now appears that a gain of another factor of five through such things as usage of parabolic light collecting mirrors, etc. is still necessary to have a reliable working spectroscopic ellipsometry system in the near IR regime. The complete system is shown schematically in fig. 5.

F. ULTRA HIGH VACUUM SYSTEM FOR ELLIPSOMETRY:

The reliability of interpretation of spectroscopic ellipsometry data and its potential ability to provide with sufficient accuracy the complex dielectric function of ultra thin layers as involved in quantum well structures depends sensitively on the cleanliness of the surface of the structure under investigation. Consequently, funds were requested and provided under this grant to develop an appropriate ultra-high vacuum (UHV) system compatible with ellipsometric system and chemical cleaning of the sample surface in an inert atmosphere prior to loading into the UHV system. Such a system had to be custom designed, keeping in mind the technical requirements and the degree of complexity and the willingness of an appropriate UHV vendor to fabricate such a system at an acceptable cost.

Better part of a year starting fall of 1983 was spent designing and obtaining estimated costs from Vendors in an interactive and iterative way before arriving at a zeroth order system in late 1984. An important consideration in developing such a system was the fact that given the basic expense of the pumping system for achieving UHV conditions and sample transfer, manipulation and heating/cooling capabilities, it would have been unwise to design the UHV chamber itself for ellipsometry alone, even though it would have made the task enormously simpler. Rather, it was important to provide for and configure a variety of ports on the chamber capable of later accommodating a few of the basic surface diagnostic techniques complimentary to spectroscopic ellipsometry. The absolute need for such techniques (for example Auger spectroscopy, X-ray photoemission spectroscopy, etc.) to complement ellipsometric measurements in-situ is not only dictated by pragmatic considerations of knowing the degree of chemical cleanliness of the surface (the reason for having a UHV environment in the first place) but also for reliable scientific analysis of the data and its interpretation in terms of chemical versus physical effects. This need for

complimentary techniques had been demonstrated in our work on ion implanted Si and on the nitridation of the thermally grown amorphous  $\text{SiO}_2$  on Si. Consequently, considerable time had to be spent investigating potential candidate systems for Auger, XPS, etc. for even though these would be mounted later, their specific technical requirements of port sizes, orientation, working distances between the sample and the particular instruments, etc. had to be accounted for at the time of designing the chamber. As can be expected, such a process requires prospective vendors to provide considerable engineering information on their surface diagnostic instruments - information that vendors generally are very reluctant to provide, particularly when the instruments are not being bought but, at best, may be bought in the future. We thus had to pay an inevitable price in terms of time to obtain such information and fold it into the design of the chamber. The final UHV system was ordered in May 1985.

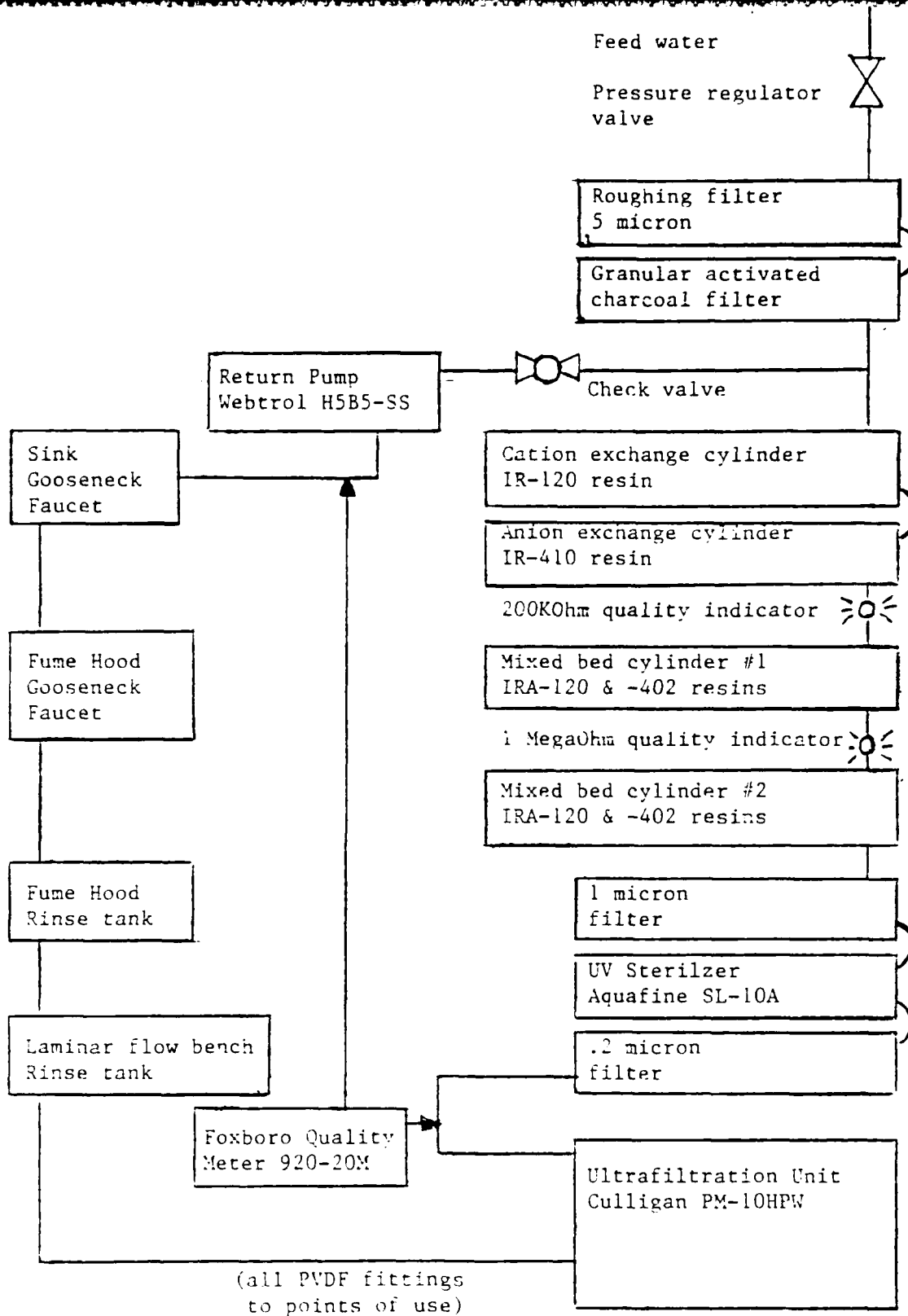
This system was to be delivered by summer of 1986. However, during the manufacturing process the vendor faced many unanticipated technical issues which required further considerations and modifications. The system delivery has thus gotten delayed and is now expected to be delivered by the beginning of 1987. It was the principal investigator's hope that in writing this final report some results on the performance of the system could be included. Indeed, the writing of this report was delayed in anticipation of fulfilling this hope but now it appears that this additional information, if needed as part of the final report, should be sent as a supplement rather than delaying the rest of the report.

#### G. ELECTRICAL MEASUREMENTS:

Finally, some components (such as electrometer, LCR-meter, etc.) of a system for such electrical measurements as the Hall mobility, C-V characteristics and I-V characteristics were acquired under the present grant. A system, based upon a Janis cryostat and an electromagnet capable of generating magnetic fields up to 5 KGauss (both already with the principal investigator), for conducting the above noted measurements was designed and put into effect. A schematic diagram of the system is shown in fig. 6. This system was further fully automated via an Apple IIe computer acquired through funds from other sources and has been in operation since fall of 1983.

## FIGURE CAPTIONS

- Fig. 1. Schematic diagram of the ultra high purity de-ionized water system, the DI water sink, fume hood and laminar flow bench arrangement for preparation of substrates and handling of chemicals.
- Fig. 2. Schematic Diagram of the pumping system and configuration for the growth chamber of the  $\Phi$ -400 MBE system.
- Fig. 3. Schematic Diagram of the RHEED intensity measurement system.
- Fig. 4. Schematic Diagram of the photoluminescence, excitation spectroscopy and optical absorption measurement system.
- Fig. 5. Schematic Diagram of the spectroscopic ellipsometry system, covering both the original UV to visible regime and the extension into the near IR regime.
- Fig. 6. Schematic Drawing of the electrical measurements system.



Points-Of-Use

Water Purification

THE CULLIGAN-ARROWHEAD ULTRA-HIGH PURITY WATER SYSTEM/U.S.C.

FIGURE 1

# PERKIN-ELMER 400 MBE SYSTEM / USC

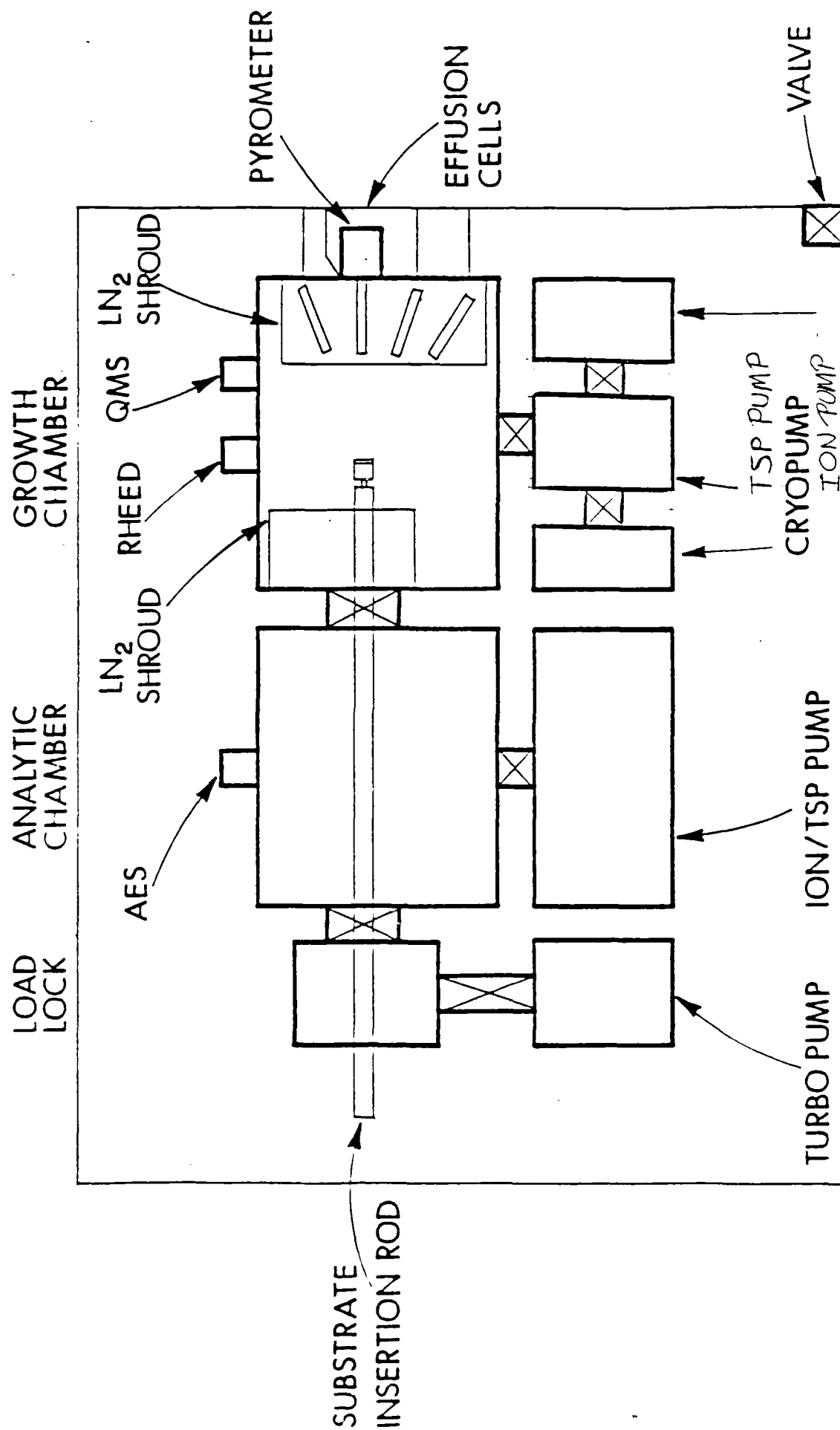
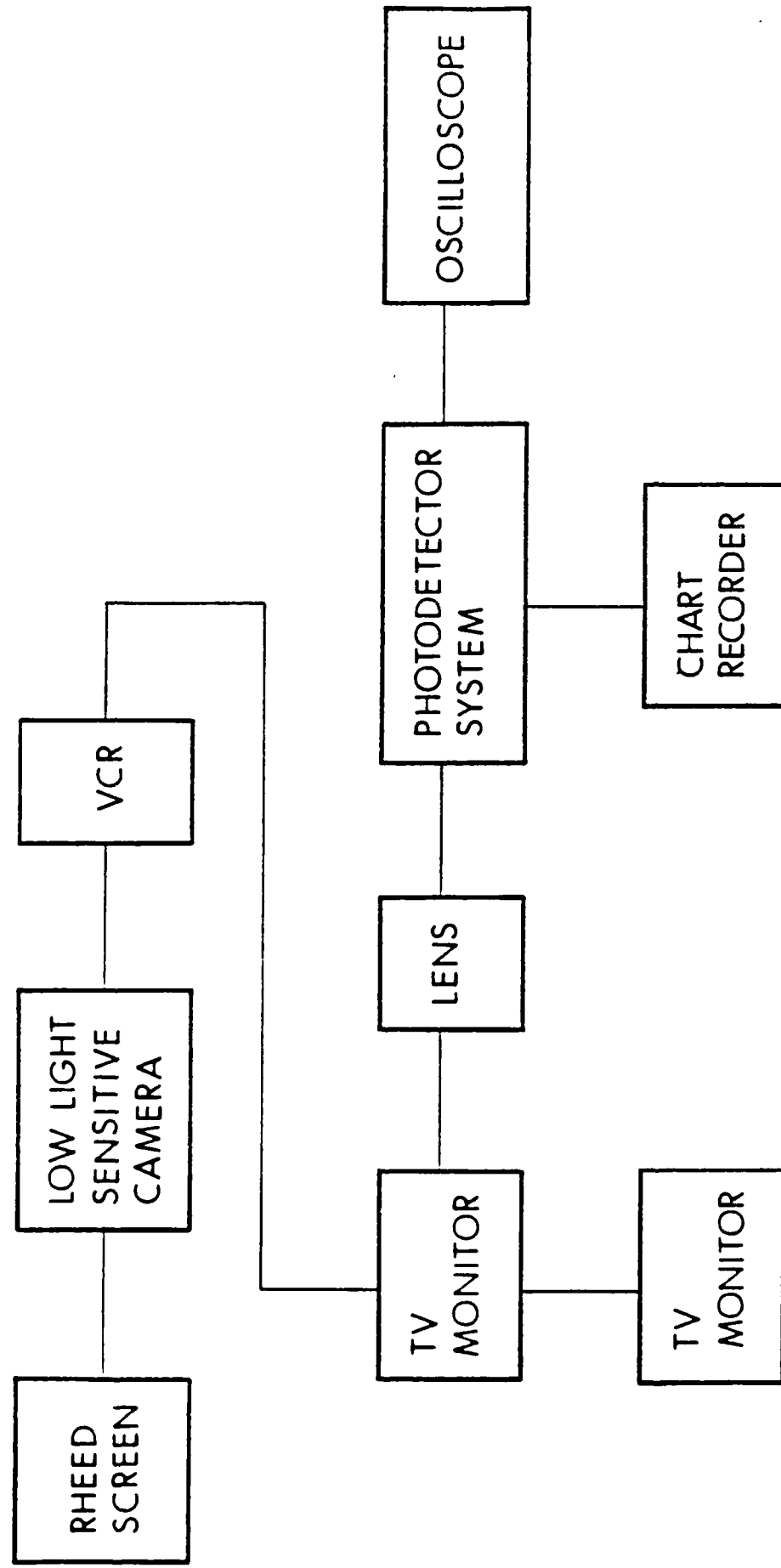
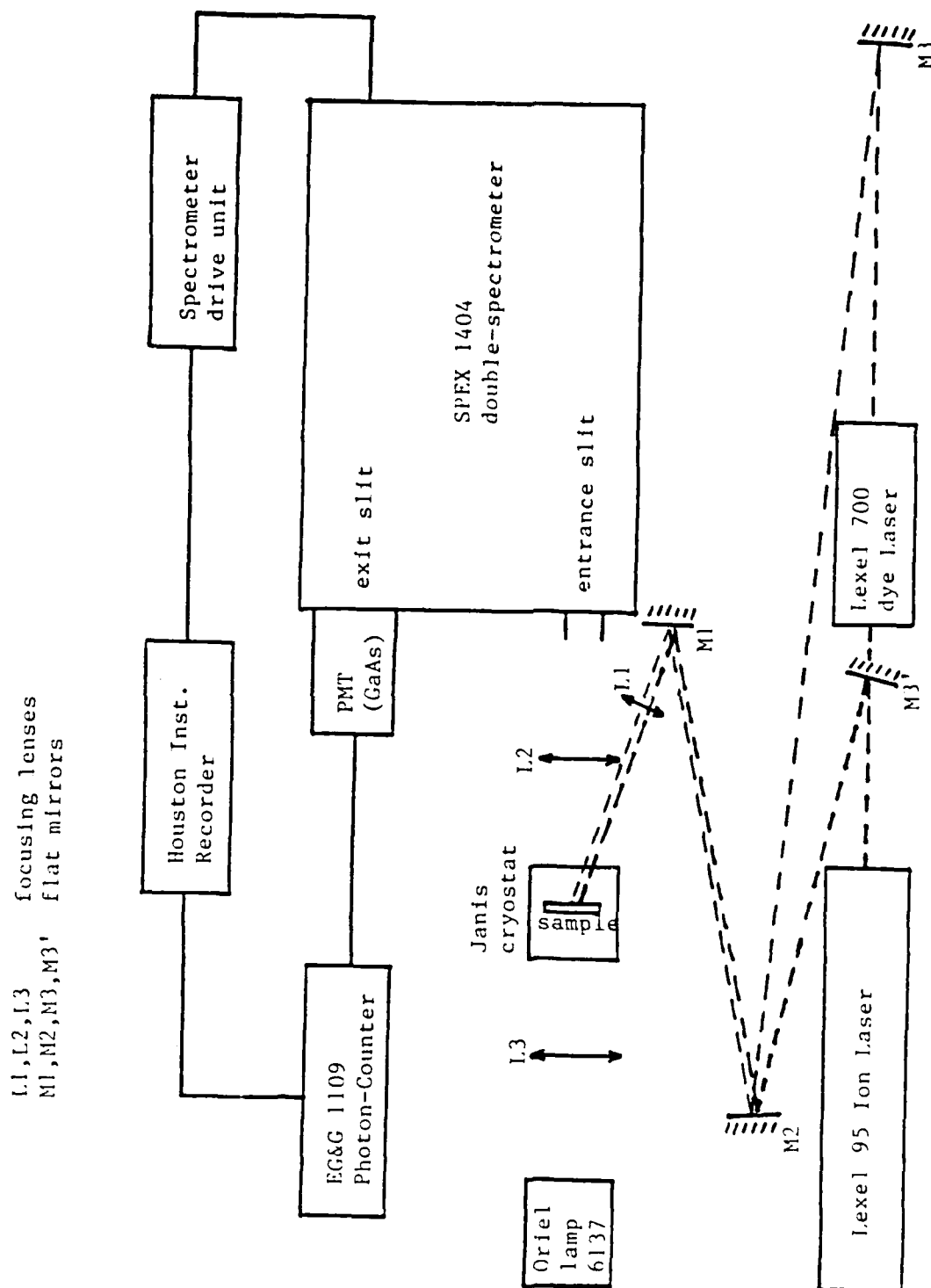


FIGURE 2



RHEED DYNAMICS MEASUREMENT SET-UP



Luminescence-Excitation Spectroscopy- Absorption Experimental Setup

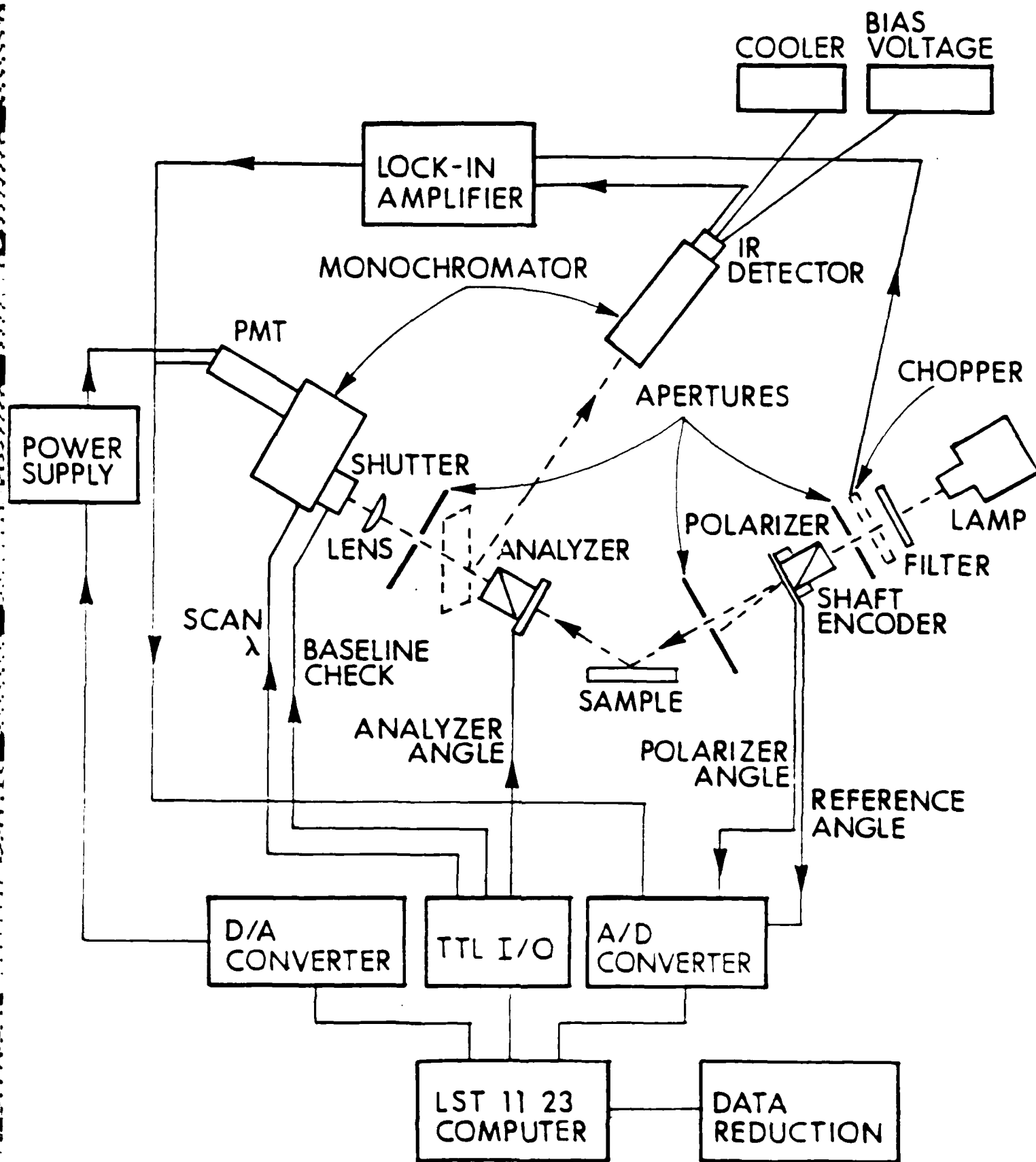


FIGURE 5



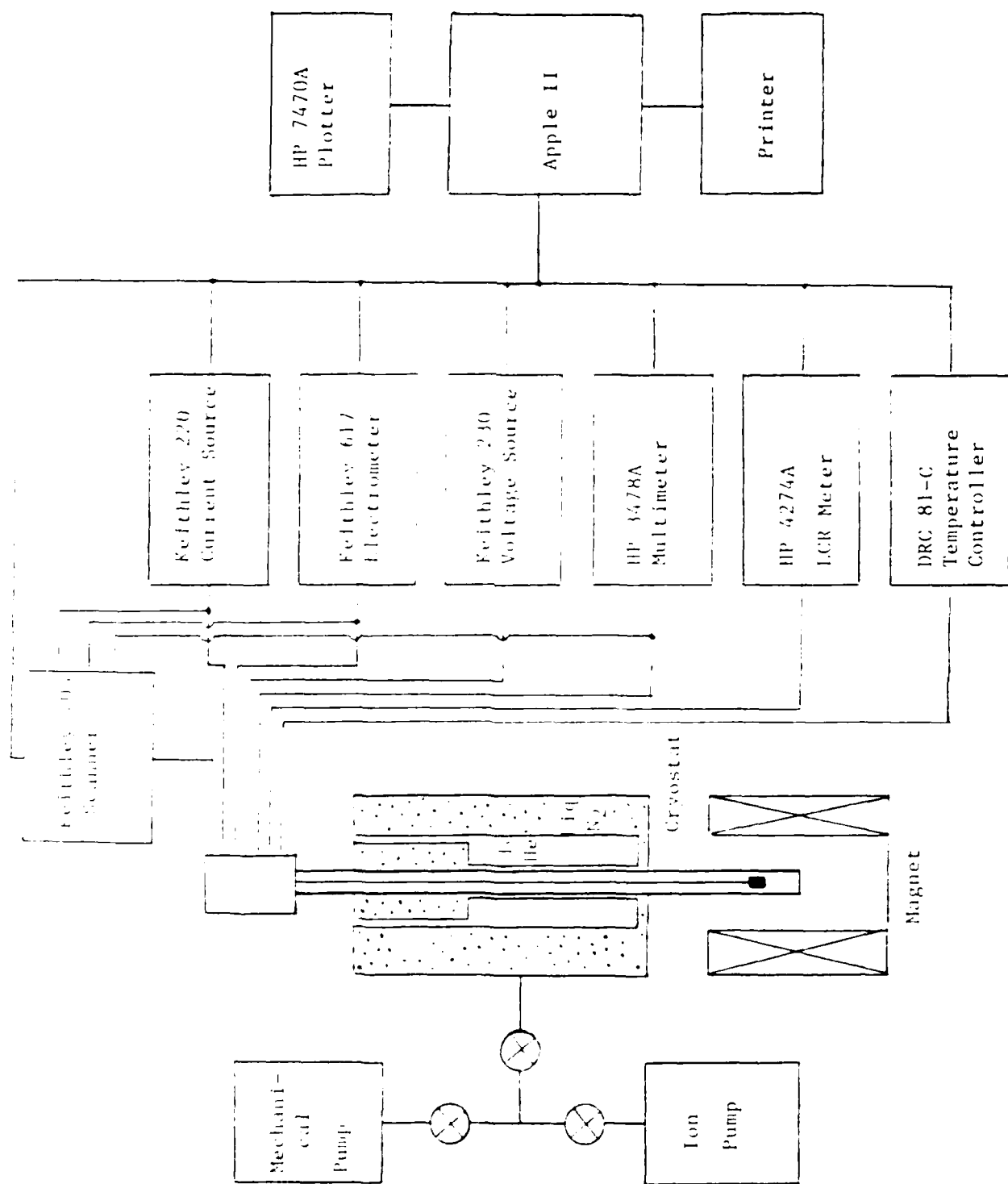


FIGURE 6

## EQUIPMENT PURCHASED

1983 - 1985

<u>SECTION</u>	<u>VENDOR</u>	<u>DESCRIPTION</u>	<u>AMOUNT</u> \$
<u>A. SUBSTRATE PREPARATION FACILITIES:</u>			
A.1.	Clean Room Products	Model 3001 Vertical Laminar Flow Work Station with exhaust	3,725
A.2	Lab. Fabricators Co.	Air-Flow Fume Hood	6,300
A.3	Culligan Water Condition. Arrowhead	UHP Water	8,107
A.4	Ryan Herco Plastics		561
A.5	Ryan Herco Plastics	PVDF Fittings	505
A.6	Terra Universal	Dessicator Cabinets	910
<u>B. MODIFICATION OF THE Ø-400 MBE SYSTEM:</u>			
B.1	Union Carbide Corp.	PBN Crucibles (2 orders)	3,024
B.2	MDC Manufact. Inc.	Flanges for Source Cells, Cryo-pumps and ion TSP Pumps	663
B.3	Thermionics Lab. Inc.	Pumping Well & Qualifications	8,518
B.4	Perkin Elmer	Ion Pump	7,545
B.5	Perkin Elmer	Digital Ion Gauge Controller	2,938
B.6	Micro-Tec West	CT8 Cryopump	9,873
B.7	V A T	8" Gate Valve	2,290
B.8	Varian Vacuum	Vacuum Fittings (2)	2,798
B.9	Angeles Valve	Butterfly Valve	442
B.10	M D C	Nipple	1,090
B.11	Perkin Elmer	Bolt Sets - Windows	1,851
B.12	Varian Vacuum Products	TI Ball Pump and Controller	4,292
B.13	Perkin Elmer	Intro Prob Rod (New & 2nd Rod)	2,130
B.14	Perkin Elmer	Repair Rod	2,609
B.15	Digital Equipment	VAX-II Computer System complete	22,104
B.16	KEPCO	16 CH D/A Dig. Programming Cage and Dual Chan. Card (Binary)	7,126
B.17	Andromeda Systems	12 Bit A/D (16 channels) (2)	1,065
B.18	Loonan Computer Products	Line printer, stand, modem and graphics terminal	3,570
B.19	Ircon	Pyrometer	3,349
B.20	Thermionics Lab	Repair Intergate Valve	880
B.21	Thermionics Lab	Electropneumatic Gate Valve for UHV System with valve position indicator	2,242

C. RHEED INTENSITY MEASUREMENT SYSTEM

C.1	Instant Replay Eqpt.	V C R Camera	2,528
C.2	Hewlett Packard	Chart Recorder	11,577
C.3	Grant Technol. Systems	Gen. Purpose Digital Board	577

D. OPTICAL MEASUREMENTS:

D.1	Janis Res. Co.	Model St-5 "Super-Tran" Cryostat with sample holders	7,125
D.2	Janis Res. Co.	Temperature Controller	4,214
D.3	ORIEL Corp. of America	6137 Universal Lamp Housing 1½ Aperture (250 w)	4,883
D.4	Keithley Instruments	617 Programmable Electrometer Source	4,429
D.5	Products for Research	Regulated High Voltage Supply	
D.6	Melles Griot	TE210TSRF Super Cooled Housing	4,204
D.7	Newport Corp.	Optical Collim. & Beam Split.Eqpt.	1,277
D.8	Cryogenics Distributors	Optical Table Mounting System	6,192
		Trans. Liquid Helium Containers	3,060

E. NEAR-IR SPECTROSCOPIC ELLIPSOMETRY:

E.1	Instruments S.A.	Interchangeable Grating Monochrom.	3,360
E.2	Thorn EMI Gen. Com.	Power Supply	495
E.3	Opto-Electronics	Sulfide Detector with Cooler	1,012
E.4	Ealing Corp.	25-5000 Hz VF Chopper	540
E.5	Andromeda Systems	4 CH 12 bit D/A Converter etc.	948
E.6	Oriel Corp.	Long Pass Filters for infrared Ellip.	870
E.7	Newport Corp.	Mirrors	856
E.8	Hamilton Electro Sales	Instrument bus. interface for LSI-II	719
E.9	Laser Precision	Variable speed light chopper	1,518

F. ULTRA HIGH VACUUM SYSTEM FOR ELLIPSOMETRY:

F.1	Microscience Inc.	Surface Science System Part II	32,407
F.2	Metro-Line Western	UTI Quad Mass Spec.	17,241

G. ELECTRICAL MEASUREMENTS:

G.1	Hewlett Packard	Voltmeter, 10CH Multiplexer scanner	8,957
		10CH Low Thermal, Relay Assy,	
		19CH. Ref. Assy with cables	
G.2	Keithley Instruments	617 Programmable Electrometer	4,861

MISCELLANEOUS BOLTS, NUTS, ETC. FOR MACHINERY 1,243

GRAND TOTAL ..... \$ 235,600

END

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